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PATENT Case No. 10225/57 (A28)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:)
Prochazka et al.)
Serial No. 10/712,768) Examiner: Fiorito
Filing Date: November 13, 2003) Group Art Unit No. 1793
For: Process to Make Rutile Pigment From Aqueous Titanium Solutions) Confirmation No. 7402

APPEAL BRIEF

Mail Stop Appeal Brief – Patents Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Dear Sirs:

Appellants submit this Appeal Brief in response to the final rejection of the pending claims mailed on May 3, 2007 and the Advisory Action mailed October 17, 2007. Appellants previously filed a Pre-Appeal Brief Request for Review on November 1, 2007 along with a Notice of Appeal. The United States Patent & Trademark Office mailed its decision on December 11, 2007 sending this case to the Board of Patent Appeals and Interferences for review. This brief is timely filed within one month from the mailing date of the Pre-Appeal Conference decision.

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This is an appeal from the final rejection of all pending claims in the Final Office Action mailed May 3, 2007. This brief is timely filed within one month of the December 11, 2007 mailing date of the Notice of Panel Decision from the Pre-Appeal Review.

I. Real Party in Interest

The real party in interest is the assignee of this application, Altair Nanomaterials, Inc. of Reno, Nevada.

II. Related Appeals and Interferences

There are no related appeals or interferences that would affect, be affected by, or have a bearing upon, the Board's decision in the present appeal.

III. Status of Claims

Claims 1-5, 9-11, 13-15, 20-36, and 40-44 are pending in this application and have been finally rejected. Appellants appeal from the rejection of 1-5, 9-11, 13, 15, 20-36, and 40-44. Claims 6-8, 12, 16-19, and 37-39 have been previously canceled. A copy of the current claims is provided in Section VIII.

IV. Status of Amendments

The last Amendment filed in this case was transmitted by the Appellants on August 28, 2007, in response to the final office action mailed on May 3, 2007. The Amendment, which sought to simplify matters for appeal by amending claim 9, was not entered according to the Advisory Action mailed on October 17, 2007.

V. Summary of Claimed Subject Matter

The invention is defined by a single independent claim, claim 1, and dependent claims, 2-5, 9-11, 13-15, 20-36, and 40-44. A general process flow sheet can be

understood by reviewing Fig. 1 and 3. The present invention relates to a low-temperature process for producing pigment-grade rutile titanium dioxide from an aqueous solution. The process requires the following sequential steps. First, an aqueous fee solution comprising a titanium compound is prepared (paras. [0031] and [0034] and Figs. 1 and 2). The titanium compound in the aqueous feed solution may be one of titanium chloride, titanium oxychloride, or mixtures thereof (para. [0034]).

Second, a catalyzing salt is added to the solution (paras. [0031] and [0036] – [0040]) in an amount from about 3% by weight of the equivalent amount to titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution (para. [0039]). Other amounts of the catalyzing salt are contemplated such as between about 10 weight % and about 50 weight % of the equivalent amount of TiO₂ present in the feed solution or between about 15 weight % and about 30 weight % of the equivalent amount of TiO₂ present in the feed solution (para. [0039]).

The catalyzing salt is selected from two or more of NaCl, KCl, and LiCl (para. [0038]) and is preferably a eutectic mixture of NaCl, KCl, and LiCl (para. [0040] and Fig. 3) but may also be a eutectic mixture of either LiCl and KCl or LiCl and NaCl (para. [0040]).

Third, a chemical control agent may optionally be added to the solution (paras. [0031] and [0042]). The chemical control agent, when added, may be added before

evaporating (para. [00440] and Fig. 2) and may include a tin compound such as tin chloride (para. [0042] and [0072]).

Fourth, the solution is evaporated to produce a dry amorphous intermediate that includes a mixture of titanium compounds (paras. [0031], [0044], and [0045]). The evaporation may be conducted in a spray drier at a temperature between about 100°C and about 300°C. (original claim 33). The amorphous intermediate may include a homogeneous mixture of titanium, oxygen, chlorine, and hydrogen compounds, with a homogeneous distribution of salts through the titanium intermediate (para. [0044]).

Finally, the intermediate is calcined to form titanium dioxide rutile pigment base (paras. [0031], [0048]- [0054]). The calcination temperature can be one of less than 800°C. or less than 700°C. or less than 500°C. or less than 500°C. or less than 400°C. and the time of calcination may be between the time needed to melt the catalyzing salt and about 24 h. or less than about two hours or less than about 30 minutes or less than about one minute (para. [0050] and [0051]). The product of calcination comprises TiO₂ rutile crystallites bound in a structure of hollow spheres or parts of spheres that may have a diameter of about 0.1 to about 100 μm. (para. [0053]).

The titanium dioxide rutile pigment base may be washed to remove the salt (paras. [0031] and [0061]). Thereafter, the washed base may be milled to disperse the pigment (paras. [0031] and [0065]).

VI. Grounds of Rejection to be Reviewed on Appeal

The issues on appeal are (1) whether there was error in the final rejection of the pending claims 1-5, 9-11, 13-15, 20-36, 40-41, and 43-44 as being obvious under 35 U.S.C. § 103(a) based on WO PCT 01/00530 to Duyvesteyn (hereinafter referred to as D1) and (2) whether there was error in the final rejection of the pending claims 1-8 [sic 5], 13-15, 20-23, 25-27, 29-36, and 40-44 as being obvious under 35 U.S.C. § 103(a) based on US 6,440,383 to Duyvesteyn (hereinafter referred to as D2).

VII. Argument

Appellants request that the Board reverse the final rejection of all the pending claims, claims 1-5, 9-11, 13-15, 20-36, and 40-44 as being obvious under 35 U.S.C. § 103(a) for at least the reason that the cited references (D1 and D2) do not teach or render obvious all the limitations of the claims. Arguments for specific claim groups and the specific issues of rejection to be addressed are presented in the separate headings below.

A. Claim 1 is patentable over D1

Claim 1 was rejected under 35 U.S.C. §103(a) as being obvious over D1. The rejection is erroneous and should be reversed. The premise of the rejection is that "it appears that it would have been obvious to form the catalyzing salt as a mixture of two or more of NaCl, KCl, and LiCl since D1 teaches that minor quantities of chemical control agents may be introduced into the titanium chloride solution to control the physical and mineralogical characteristics of the solid titanium dioxide product." (page 3, Final Rejection mailed May 3, 2007).

D1 does not recognize the use of a catalyzing salt that does not significantly change chemical composition, does not recognize using a catalyzing salt that includes at least two of NaCl, KCl, and LiCl, and does not recognize using an amount of the catalyzing salt in an amount from about 3% by weight of the equivalent amount of titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution.

At best, D1 teaches only that minor quantities may be added, but there is no mention in D1 about what constitutes a minor quantity. Moreover, D1 does not mention that it is the aggregate of the two or more of NaCl, KCl, and LiCl that must be present in an amount from about 3% by weight of the equivalent amount of titanium dioxide. In the absence of any showing of these limitations in D1, the rejection cannot stand.

As explained in the specification, the use of two or more of NaCl, KCl, and LiCl in an amount from about 3% by weight of the equivalent amount of titanium dioxide promotes the formation of unstable TiO₂ crystal intermediates without chemically reacting to a significant extent with TiO₂ and, as a result, unstable crystal configurations of TiO₂ such as brookite, can form and be converted into the rutile TiO₂ phase at much lower temperatures (para. [0006]). Moreover, Fig. 6a, which is an SEM, shows that the calcination process reorganized the amorphous thin film of the intermediate to an open network of well-developed rutile crystals. As explained in the specification, it is believed that the catalyzing salts most likely improve mass transfer, provide spacing required for rutile growth to the right particle size in an open network macrostructure, and create ideal

conditions for wetting and fusing in the crystal growth process, especially after they melt (para. [0055]). Despite the advantages achieved by the claimed process, as pointed out in the specification, the Examiner nevertheless concludes that "it is well settled that determination of optimum values of cause effective variables such as these process parameters is within the skill of one practicing in the art" citing to *In re Boesch*, 205 USPQ 215 (CCPA 1980).

In re Boesch, however, is not applicable to the present situation because the facts in In re Boesch differ from those in the present application. In In re Boesch the claim was a composition claim (not a process claim) and it related to a nickel base alloy having a composition consisting essentially of certain claimed ranges of constituents such as carbon, cobalt, and others. The prior art used to reject the claim disclosed nickel based alloys containing the **same** constituents in amounts where each of the ranges of constituents in the claimed alloys overlapped ranges disclosed by the prior art. The court upheld the rejection because "the composition requirements of the claims and the cited references overlap" such that "the prior art would have suggested the kind of experimentation necessary to achieve the claimed composition" and noted that the conclusion accorded "with the rule that "discovery of an optimum value of a result effective variable in a known process is ordinarily within the skill of the art". Id. at 219.

In the present case, there is no overlap because D1 does not recognize the use of a catalyzing salt that does not significantly change chemical composition, does not

recognize using catalyzing salt that includes at least two of NaCl, KCl, and LiCl, and does not recognize using an amount of the catalyzing salt in an amount from about 3% by weight of the equivalent amount of titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution. When considering these many differences, one would not consider the facts of *In re Boesch* to be analogous to the present situation. Because the premise fails, the conclusion necessarily must fail.

B. Claims 2-5, 9-11, 13-15, 20-36, 40-41, and 43-44 are patentable over D1

Claims 2-5, 9-11, 13-15, 20-36, 40-41, and 43-44 depend from claim 1 and further limitations that are not suggested or taught by D1.

i. Claims 2, 3, and 40 are patentable over D1

Claims 3 and 40, each of which depends from claim 2 requires washing, milling, and dispersing the rutile pigment. As the Examiner acknowledges, however, D1 requires milling and then washing, which is opposite the process required by the present claim. Accordingly, D1 does not teach or suggest the process steps required by claim 3. Moreover, the Examiner has not articulated any basis why one of skill in the art would reverse the milling and washing steps of D1. Therefore, a *prima facie* case of obviousness has not been established.

ii. Claims 9-11, and 44 are patentable over D1

With respect to claims 9-11 and 44, there is simply no suggestion in D1 or elsewhere to use the claimed eutectic mixture. As noted above, the unobvious advantages resulting from the use of a eutectic mixture have been explained above and

in the specification. These surprising and unexpected advantages are not taught or appreciated by D1.

Moreover, claims 9-11 were not rejected in view of D2, which contains substantially the same disclosure (or lack thereof with respect to the claimed eutectic mixture). The lack of a rejection of claims 9-11 in view of D2 implicitly means that claims 9-11 are patentable over D1.

iii. Claims 20-24 are patentable over D1

Claims 20-24 define the calcination temperature as less than 800°C., less than 700°C., less than 600°C., less than 500°C. or less than 400°C., respectively. As an initial matter, D1 does not suggest the use of any calcination temperature less than 400°C. In addition, the rejection provides no basis for rejecting this claim and thus, it cannot stand. As for claims 20-23, because D1 does not teach or suggest all the features of claim 1, from which claims 20-23 depend, claims 20-23 are patentable.

Moreover, the fact that claim 24 was not rejected in view of D2 demonstrates the patentability in view of D1 since their disclosures (or lack thereof) with respect this limitation are substantially the same.

iv. Claims 25-28 are patentable over D1

Claims 25-28 define the time of calcination as the time of calcination between the time needed to melt the catalyzing salt and about 24 h., less than about two hours, less than about 30 minutes, or less than about one minute, respectively. As an initial matter, the final rejection does not mention a calcination time of either less than about

30 minutes or less than about one minute, as required by claims 27 and 28. Without any mention of these limitations, a *prima facie* case is not established. As for claims 25 and 26, because D1 does not teach or suggest all the features of claim 1, from which claims 25 and 26 depend, claims 25 and 26 are patentable.

Moreover, the fact that claim 28 was not rejected in view of D2 demonstrates the patentability in view of D1 since their disclosures (or lack thereof) with respect this limitation are substantially the same.

v. Claims 30 and 31 are patentable over D1

As for claims 30 and 31, while D1 discloses the use of SnCl₂•2H₂O in an amount to provide 6.3% Sn as wt% in TiO₂, the SnCl₂•2H₂O is not one of the claimed chloride salts and it significantly changes its chemical composition contrary to the limitations required by claim 1. As pointed out in the Response filed February 1, 2007, SnCl₂•2H₂O significantly changes its chemical composition. The Merck Index notes that SnCl₂•2H₂O in the presence of much water (which would be expected in the claimed aqueous fee solution), it forms an insoluble basic salt. Therefore, D1 teaches away from the claimed process.

C. Claim 1 is patentable over D2

Claim 1 was rejected under 35 U.S.C. §103(a) as being obvious over D2. The rejection is erroneous and should be reversed. The premise of the rejection is that "it appears that it would have been obvious to form the catalyzing salt as a mixture of two or more of NaCl, KCl, and LiCl since D2 teaches that minor quantities of chemical control

agents may be introduced into the titanium chloride solution to control the physical and mineralogical characteristics of the solid titanium dioxide product." (page 5, Final Rejection mailed May 3, 2007).

D2 does not recognize the use of a catalyzing salt that does not significantly change chemical composition, does not recognize using a catalyzing salt that includes at least two of NaCl, KCl, and LiCl, and does not recognize using an amount of the catalyzing salt in an amount from about 3% by weight of the equivalent amount of titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution.

At best, D2 teaches only that minor quantities may be added, but there is no mention in D2 about what constitutes a minor quantity. Moreover, D2 does not mention that it is the aggregate of the two or more of NaCl, KCl, and LiCl that must be present in an amount from about 3% by weight of the equivalent amount of titanium dioxide. In the absence of any showing of these limitations in D2, the rejection cannot stand.

As explained in the specification, the use of two or more of NaCl, KCl, and LiCl in an amount from about 3% by weight of the equivalent amount of titanium dioxide promotes the formation of unstable TiO₂ crystal intermediates without chemically reacting to a significant extent with TiO₂ and, as a result, unstable crystal configurations of TiO₂ such as brookite, can form and be converted into the rutile TiO₂ phase at much lower temperatures (para. [0006]). Moreover, Fig. 6a, which is an SEM, shows that the calcination process reorganized the amorphous thin film of the intermediate to an open

network of well-developed rutile crystals. As explained in the specification, it is believed that the catalyzing salts most likely improve mass transfer, provide spacing required for rutile growth to the right particle size in an open network macrostructure, and create ideal conditions for wetting and fusing in the crystal growth process, especially after they melt (para. [0055]). Despite the advantages achieved by the claimed process, as pointed out in the specification, the Examiner nevertheless concludes that "it is well settled that determination of optimum values of cause effective variables such as these process parameters is within the skill of one practicing in the art" citing to *In re Boesch*, 205 USPQ 215 (CCPA 1980).

In re Boesch, however, is not applicable to the present situation because the facts in In re Boesch differ from those in the present application. In In re Boesch the claim was a composition claim (not a process claim) and it related to a nickel base alloy having a composition consisting essentially of certain claimed ranges of constituents such as carbon, cobalt, and others. The prior art used to reject the claim disclosed nickel based alloys containing the **same** constituents in amounts where each of the ranges of constituents in the claimed alloys overlapped ranges disclosed by the prior art. The court upheld the rejection because "the composition requirements of the claims and the cited references overlap" such that "the prior art would have suggested the kind of experimentation necessary to achieve the claimed composition" and noted that the conclusion accorded "with the rule that "discovery of an optimum value of a

result effective variable in a known process is ordinarily within the skill of the art". *Id.* at 219.

In the present case, there is no overlap because D2 does not recognize the use of a catalyzing salt that does not significantly change chemical composition, does not recognize using catalyzing salt that includes at least two of NaCl, KCl, and LiCl, and does not recognize using an amount of the catalyzing salt in an amount from about 3% by weight of the equivalent amount of titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution. When considering these many differences, one would not consider the facts of *In re Boesch* to be analogous to the present situation. Because the premise fails, the conclusion necessarily must fail.

D. Claims 2-8 [sic 5], 13-15, 20-23, 25-27, 29-36, and 40-44 are patentable over D2

Claims 2-8 [sic 5], 13-15, 20-23, 25-27, 29-36, and 40-44_depend from claim 1 and further limitations that are not suggested or taught by D2.

i. Claims 2, 3, and 40 are patentable over D2

Claims 3 and 40, each of which depends from claim 2 requires washing, milling, and dispersing the rutile pigment. As the Examiner acknowledges, however, D2 requires milling and then washing, which is opposite the process required by the present claim. Accordingly, D2 does not teach or suggest the process steps required by claim 3. Moreover, the Examiner has not articulated any basis why one of skill in the art would reverse the milling and washing steps of D2. Therefore, a *prima facie* case of obviousness has not been established.

ii. Claims 20-23 are patentable over D2

Claims 20-23 define the calcination temperature as less than 800°C., less than 700°C., less than 600°C., or less than 500°C., respectively. Because D2 does not teach or suggest all the features of claim 1, from which claims 20-23 depend, claims 20-23 are patentable.

iii. Claims 25-27 are patentable over D2

Claims 25-27 define the time of calcination as the time of calcination between the time needed to melt the catalyzing salt and about 24 h., less than about two hours, or less than about 30 minutes, respectively. As an initial matter, the final rejection does not mention a calcination time of less than about 30 minutes, as required by claim 27. Without any mention of this limitation, a *prima facie* case is not established. As for claims 25 and 26, because D1 does not teach or suggest all the features of claim 1, from which claims 25 and 26 depend, claims 25 and 26 are patentable.

iv. Claims 30 and 31 are patentable over D2

As for claims 30 and 31, while D2 discloses the use of SnCl₂•2H₂O in an amount to provide 6.3% Sn as wt% in TiO₂, the SnCl₂•2H₂O is not one of the claimed chloride salts and it significantly changes its chemical composition contrary to the limitations required by claim 1. As pointed out in the Response filed February 1, 2007, SnCl₂•2H₂O significantly changes its chemical composition. The Merck Index notes that SnCl₂•2H₂O in the presence of much water (which would be expected in the

claimed aqueous fee solution), it forms an insoluble basic salt. Therefore, D2 teaches away from the claimed process.

v. Claim 44 is patentable over D2

As an initial matter, there is simply no suggestion in D2 or elsewhere to use the claimed eutectic mixture. As noted above, the unobvious advantages resulting from the use of a eutectic mixture have been explained above and in the specification. These surprising and unexpected advantages are not taught or appreciated by D2.

Moreover, the fact that claims 9-11, which require a eutectic mixtures, were not rejected in view of D2, demonstrates the patentability of claim 44.

E. Conclusion

In view of the above remarks, Appellants submit that the claimed invention is patentable in view of the cited references of record. Appellants therefore request reversal of the rejections of Claims 1-5, 9-11, 13-15, 20-36, and 40-44.

Respectfully submitted,

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VIII. Claims Appendix

- 1. (Previously Presented) A low-temperature process for producing pigment-grade rutile titanium dioxide from an aqueous solution comprising the following sequential steps:
 - a. preparing an aqueous feed solution comprising a titanium compound;
- b. adding a catalyzing salt selected from two or more of NaCl, KCl, and LiCl to the solution in an amount from about 3% by weight of the equivalent amount of titanium dioxide to about the amount corresponding to the saturation point of the catalyzing salt in the feed solution, wherein the catalyzing salt does not significantly change chemical composition;
 - c. optionally adding a chemical control agent to the solution;
- d. evaporating the solution to produce a dry amorphous intermediate that includes a mixture of titanium compounds; and
- e. calcining the intermediate to form TiO₂ phase pure rutile pigment base at a temperature greater than a melting point temperature of the catalyzing salt and for a period of time sufficient to melt the catalyzing salt.
- 2. (Original) The process of claim 1 further comprising washing the salt from the calcined TiO₂ rutile pigment base.
- 3. (Original) The process of claim 2 further comprising milling and dispersing the TiO₂ rutile pigment.

- 4. (Original) The process of claim 1 wherein the titanium compound is selected from the group of titanium chloride, titanium oxychloride, and mixtures thereof.
- 5. (Original) The process of claim 1 wherein the titanium compound is titanium oxychloride.
- 6. (Canceled)
- 7. (Canceled)
- 8. (Canceled)
- 9. (Previously presented) The process of claim 1 wherein the catalyzing salt is a eutectic mixture of two or more of NaCl, KCl, and LiCl.
- 10. (Previously presented) The process of claim 1 wherein the catalyzing salt is a eutectic mixture of LiCl and KCl.
- 11. (Previously presented) The process of claim 1 wherein the catalyzing salt is a eutectic mixture of LiCl and NaCl.
- 12. (Canceled)
- 13. (Original) The process of claim 1 wherein the catalyzing salt present in the feed solution is from about 10 weight % and about 50 weight % of the equivalent amount of TiO₂ present in the feed solution.

- 14. (Original) The process of claim 1 wherein the amount of the catalyzing salt is between about 15 weight % and about 30 weight % of the equivalent amount of TiO₂ present in the feed solution.
- 15. (Original) The process of claim 1 wherein the catalyzing salt does not significantly chemically react with titanium oxide through the process.
- 16. (Canceled)
- 17. (Canceled)
- 18. (Canceled)
- 19. (Canceled)
- 20. (Original) The process of claim 1 wherein the calcining is conducted at a temperature less than 800°C.
- 21. (Original) The process of claim 1 wherein the calcining is conducted at a temperature less than 700°C.
- 22. (Original) The process of claim 1 wherein the calcining is conducted at a temperature less than 600°C.
- 23. (Original) The process of claim 1 wherein the calcining is conducted at a temperature less than 500°C.

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- 24. (Original) The process of claim 1 wherein the calcining is conducted at a temperature less than 400°C.
- 25. (Original) The process of claim 1 wherein the calcination time is between the time needed to melt the catalyzing salt and about 24 h.
- 26. (Original) The process of claim 1 wherein the calcination time is less than about two hours.
- 27. (Original) The process of claim 1 wherein the calcination time is less than about 30 minutes.
- 28. (Original) The process of claim 1 wherein the calcination time for is less than about one minute.
- 29. (Original) The process of claim 1 wherein the chemical control agent is added before evaporating.
- 30. (Previously presented) The process of claim 29 wherein the chemical control agent is a tin compound.
- 31. (Previously presented) The process of claim 29 wherein the chemical control agent is tin chloride.
- 32. (Original) The process of claim 1 wherein the evaporating is conducted in a spray drier.

- 33. (Original) The process of claim 1 wherein the evaporation temperature is between about 100°C and about 300°C.
- 34. (Original) The process of claim 1 wherein the amorphous intermediate comprises a homogeneous mixture of titanium, oxygen, chlorine, and hydrogen compounds, with a homogeneous distribution of salts through the titanium intermediate.
- 35. (Original) The process of claim 32 wherein the product after calcination comprises TiO₂ rutile crystallites bound in a structure of hollow spheres or parts of spheres.
- 36. (Previously presented) The process of claim 35 wherein the spheres have a diameter of about 0.1 to about 100 μ m.
- 37. (Canceled)
- 38. (Canceled)
- 39. (Canceled)
- 40. (Previously presented) The process of claim 2 wherein the washing is conducted with water to provide an aqueous salt solution and the TiO₂ rutile pigment base.
- 41. (Original) The process of claim 40 further comprising recycling the salts in the aqueous salt solution.

- 42. (Original) The process of claim 41 further comprising milling the washed TiO₂ rutile pigment base product.
- 43. (Original) The process of claim 1 wherein a thermodynamically unstable brookite phase is formed as an intermediate during the early stages of calcination.
- 44. (Previously presented) The process of claim 1 wherein the catalyzing salt is a eutectic mixture of NaCl, KCl, and LiCl.

IX. Evidence Appendix

N/A

X. Related Proceedings Appendix

N/A